BENEATH THE SURFACE: X-RAY ANALYSES OF BATTERY MATERIALS AND STRUCTURES

A Battery Webinar Series by Rigaku

Non-destructive Elemental Analysis of Batteries Using XRF

Starting at 1 pm CDT

- *You will be muted during the webinar.*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working.*

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A Battery Webinar Series by Rigaku

Non-destructive Elemental Analysis of Batteries Using XRF

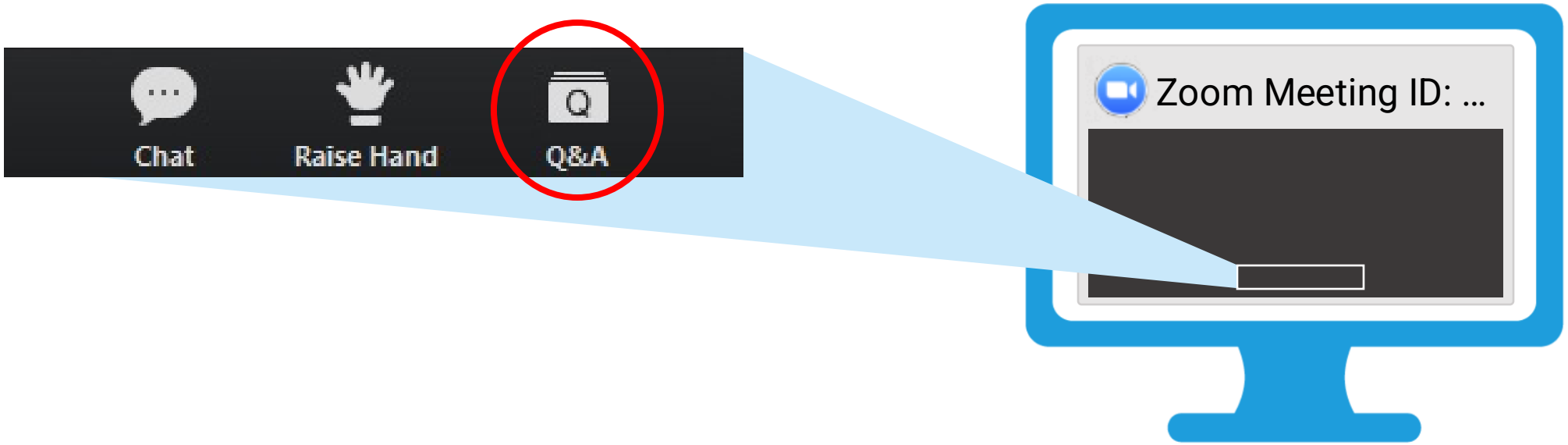
Starting at 1 pm CDT

We are starting now…

Presenter: **Amber Quevy** | XRF Applications Lab Manager

Co-presenter: **Tim Bradow** | Sr. Business Development Manager

Host: **Aya Takase** | Head of Global Marketing



You can ask questions during the presentation.
Please use the Q&A to ask questions.

Recording will be available tomorrow.

Non-Destructive Elemental Analysis of Batteries Using XRF

You will learn

1. **What** is XRF?
2. **Why** use XRF?
3. **Where** is XRF used?
4. **How** is XRF used? **Examples!**

Polling Question #1

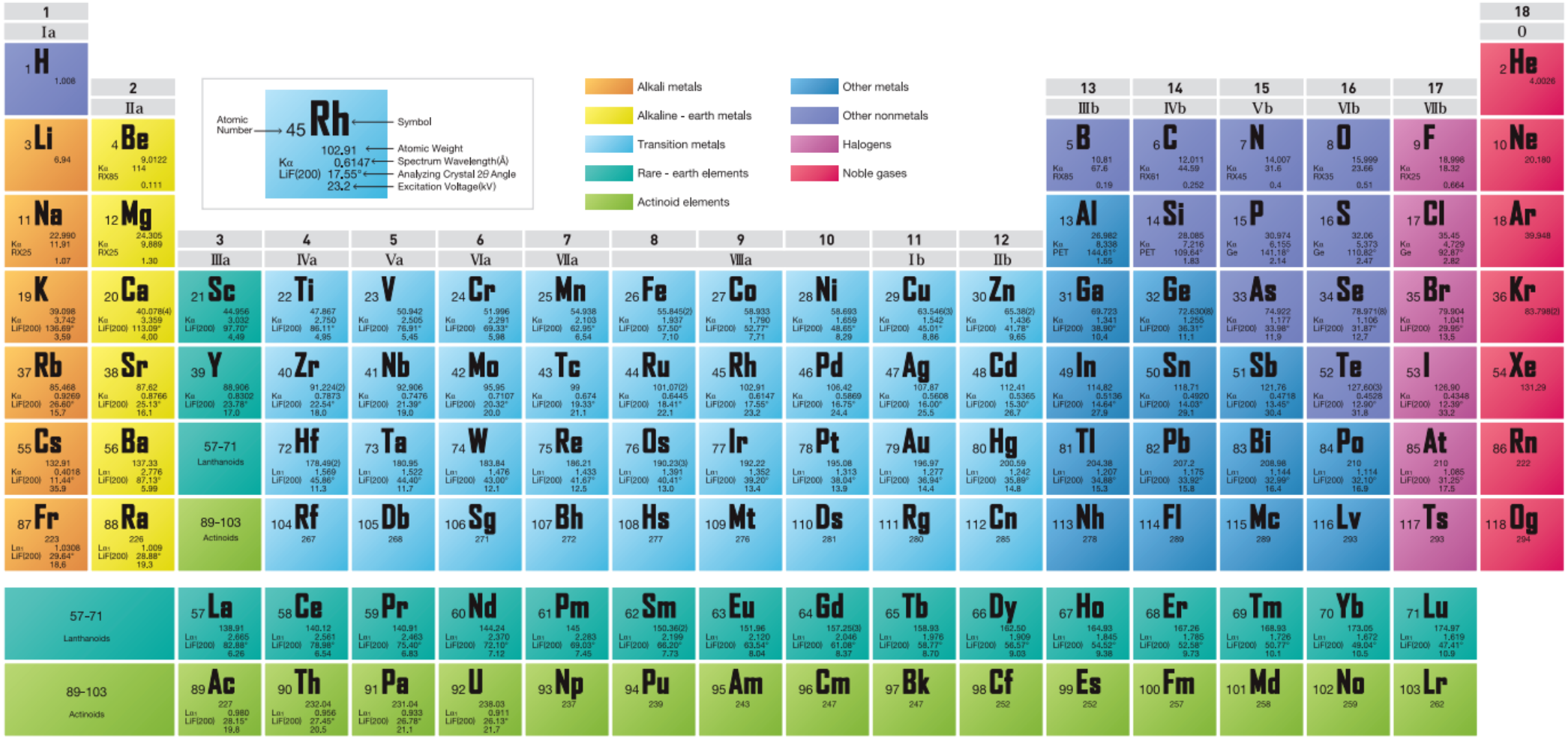
Microsoft Stock

What is XRF?

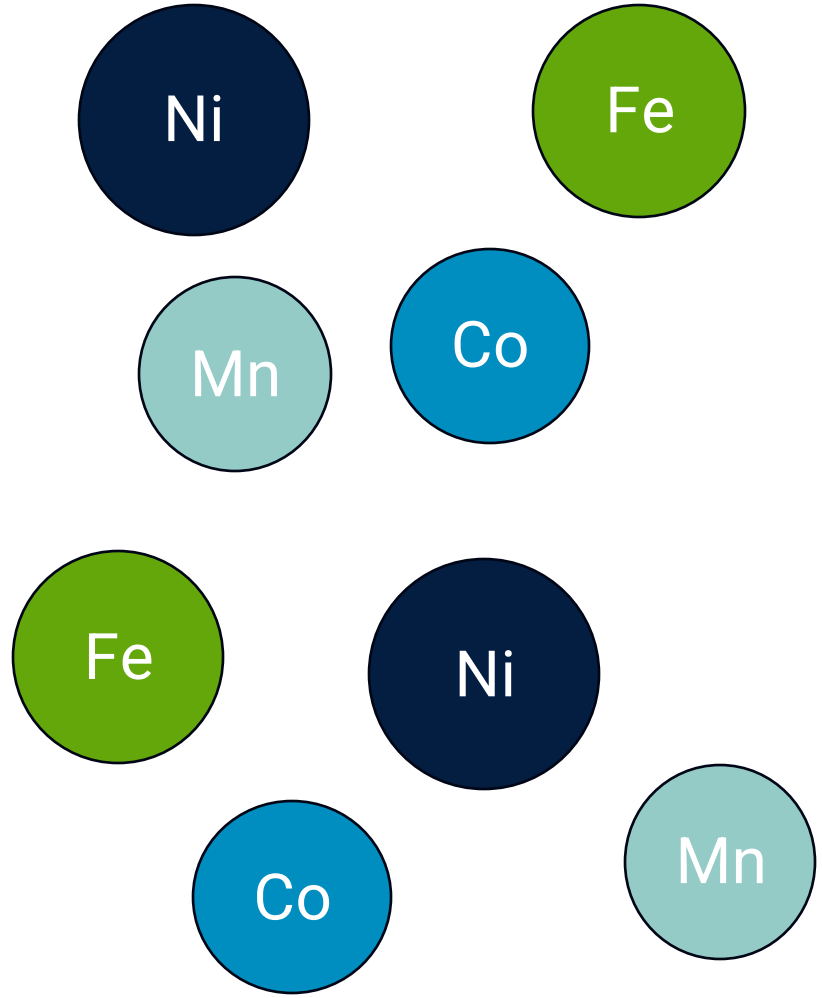
The Subject

The Building Blocks

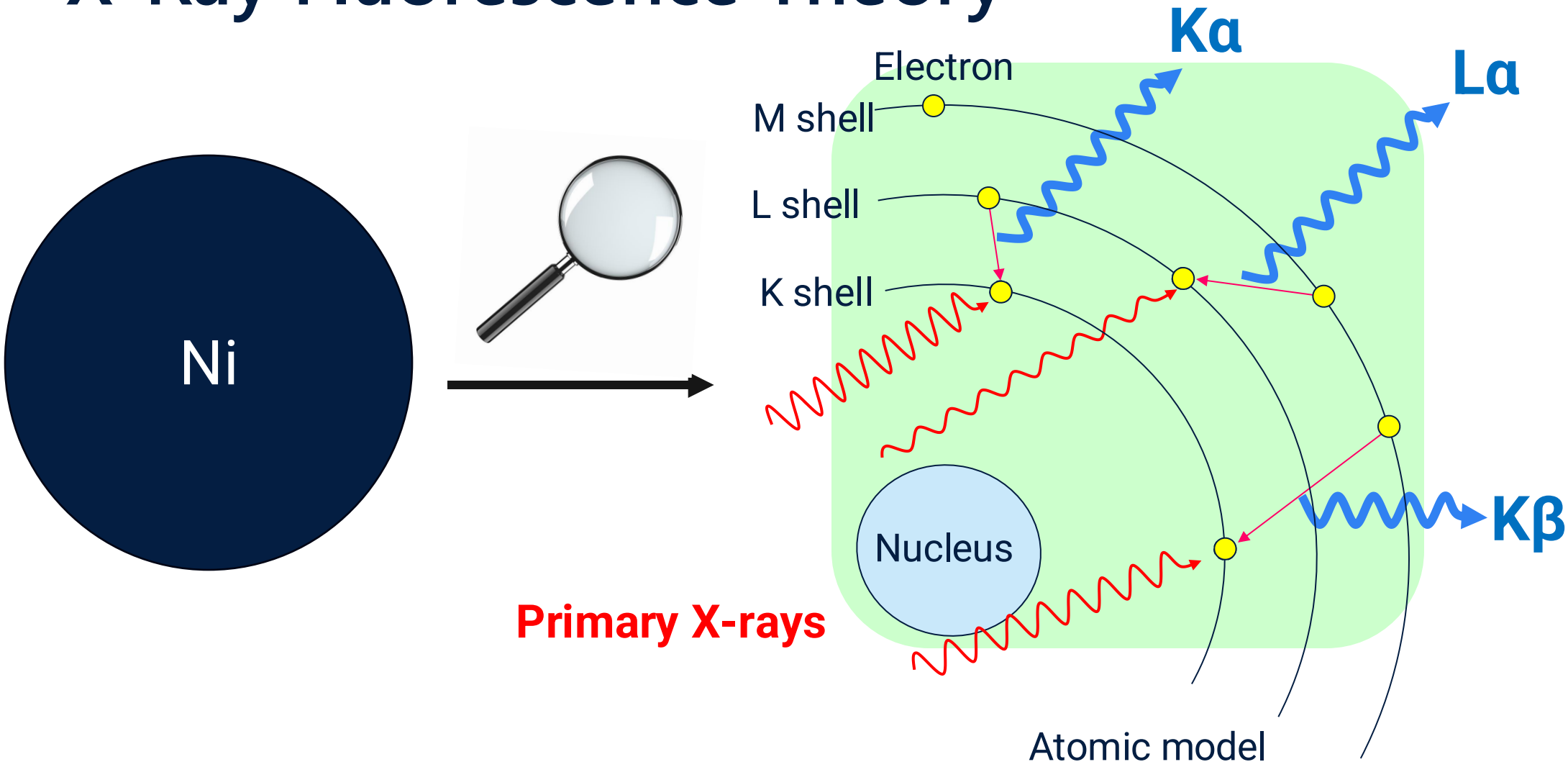
Periodic Table of Chemical Elements



Bob's Close-up



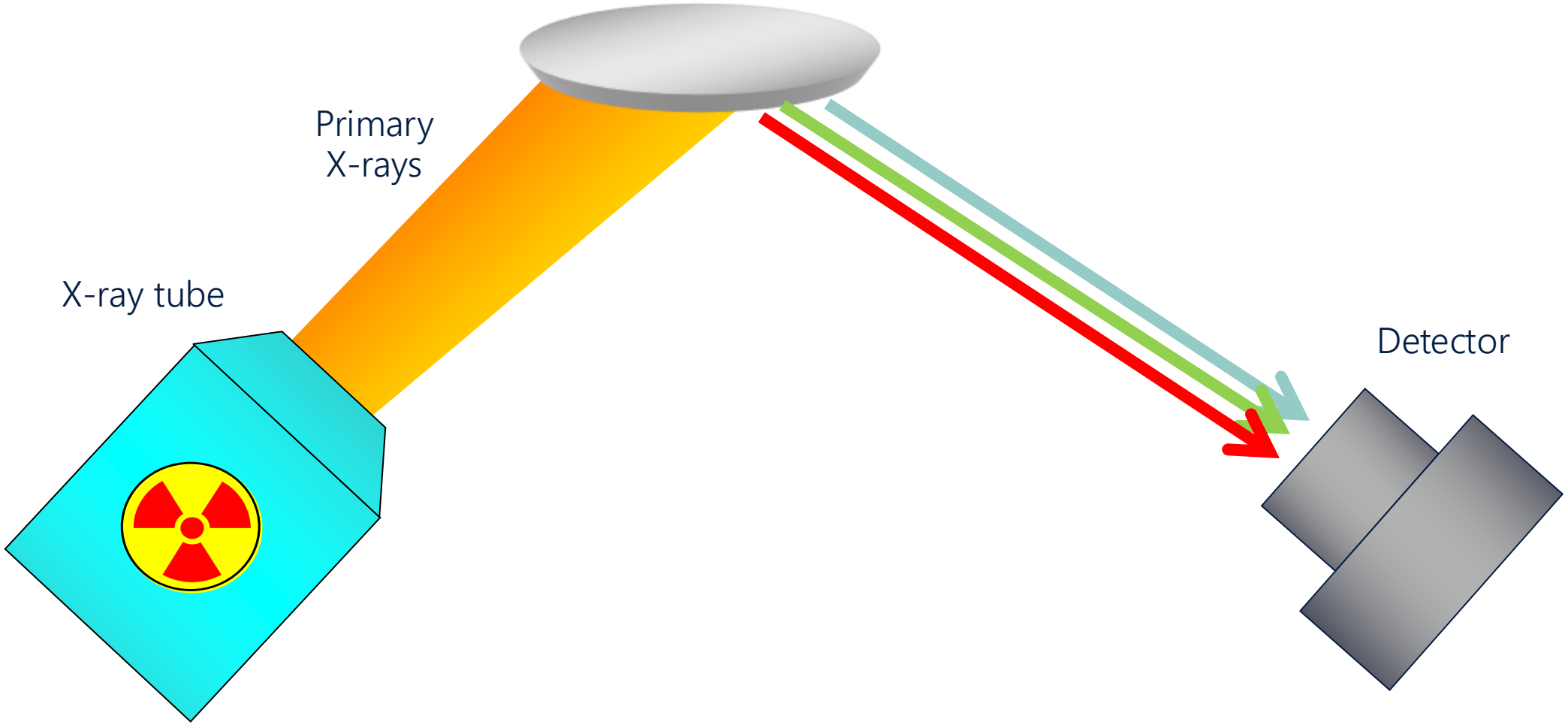
X-Ray Fluorescence Theory



X-ray Fluorescence Types: EDXRF vs. WDXRF

Instrument	EDXRF	WDXRF
Format	Handheld or Benchtop	Benchtop and Floor models
Tube	Ag, Pd, Rh	Pd, Rh
Wattage	4W-100W	200W-4000W
Analysis range	Na-U	Be-Cm
Atmosphere Options	Air, Helium, Vacuum	Helium, Vacuum
Cost	$-$$	$$-$$$

Types of XRF: Energy Dispersive (EDXRF)



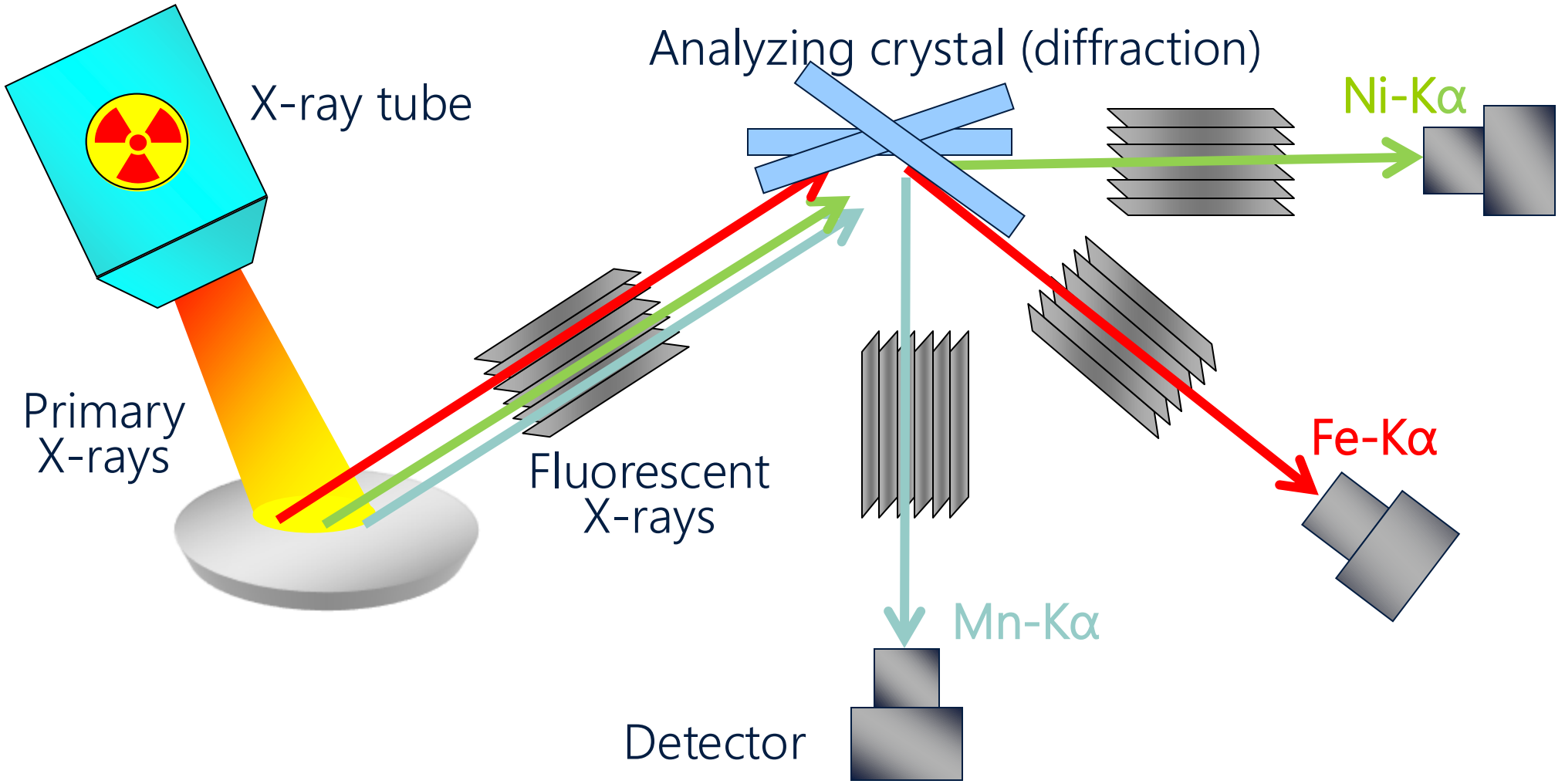
Examples of EDXRF Instruments

NEX QC
Series

NEX DE
Series

NEX CGII
Series

Types of XRF: Wavelength Dispersive (WDXRF)



Examples of WDXRF Instruments

Supermini200

ZSX Primus Series

Polling Question #2

Microsoft Stock

Why use XRF?

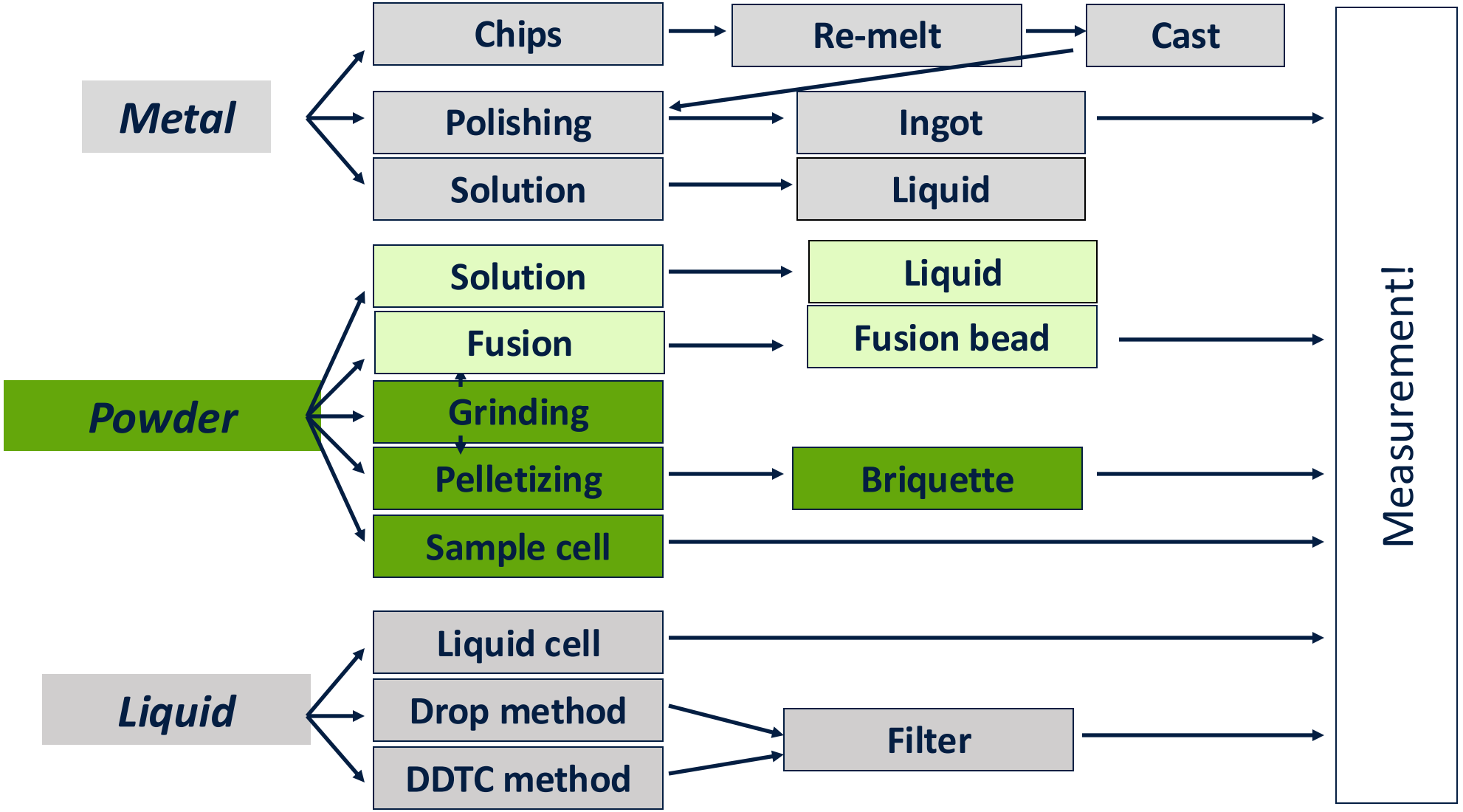
Traditional Analysis Techniques

1. Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
2. X-ray Photoelectron Spectroscopy (XPS)
3. X-ray Fluorescence (XRF)
4. Others!

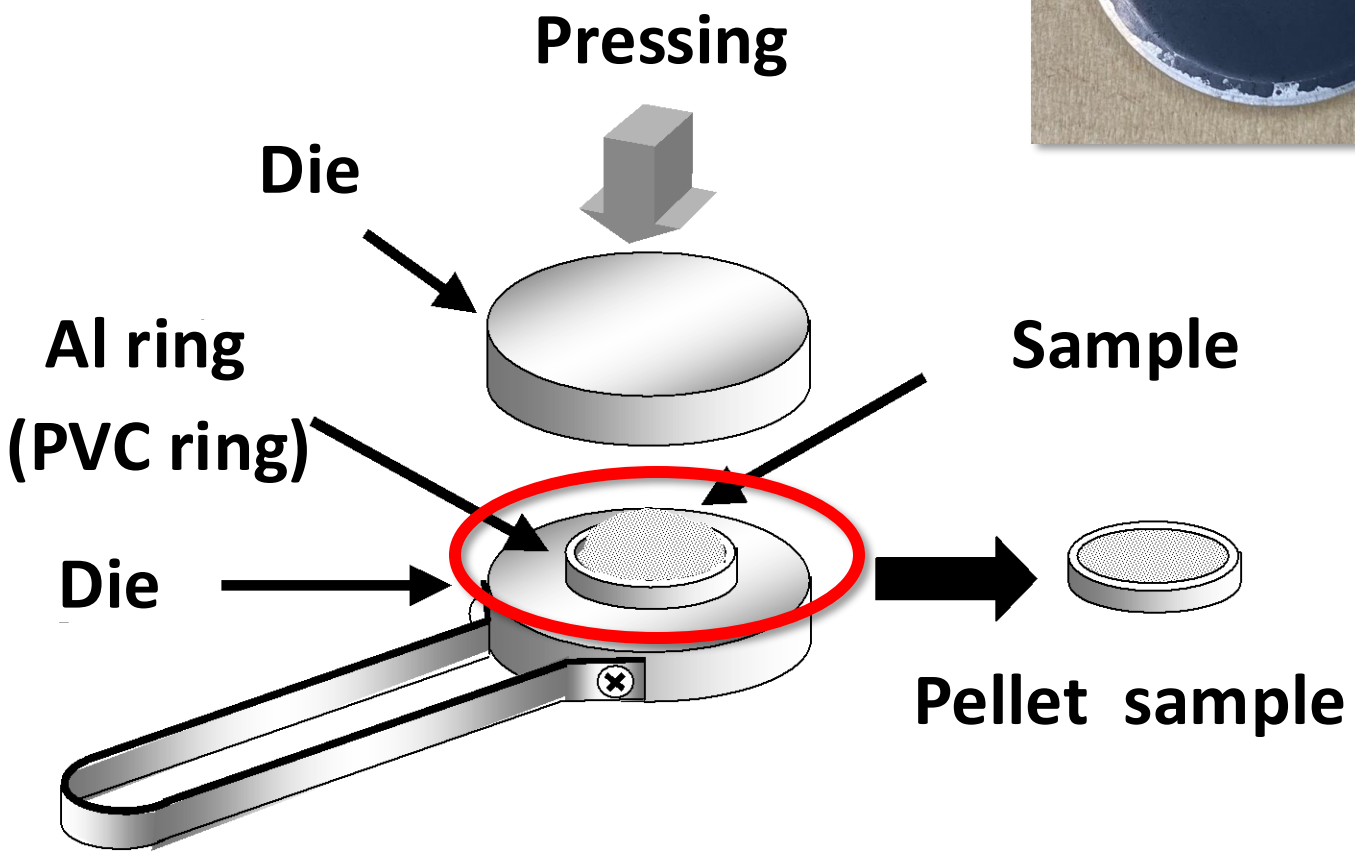
Sample Preparation

1. Solids
2. Powders
3. Liquids

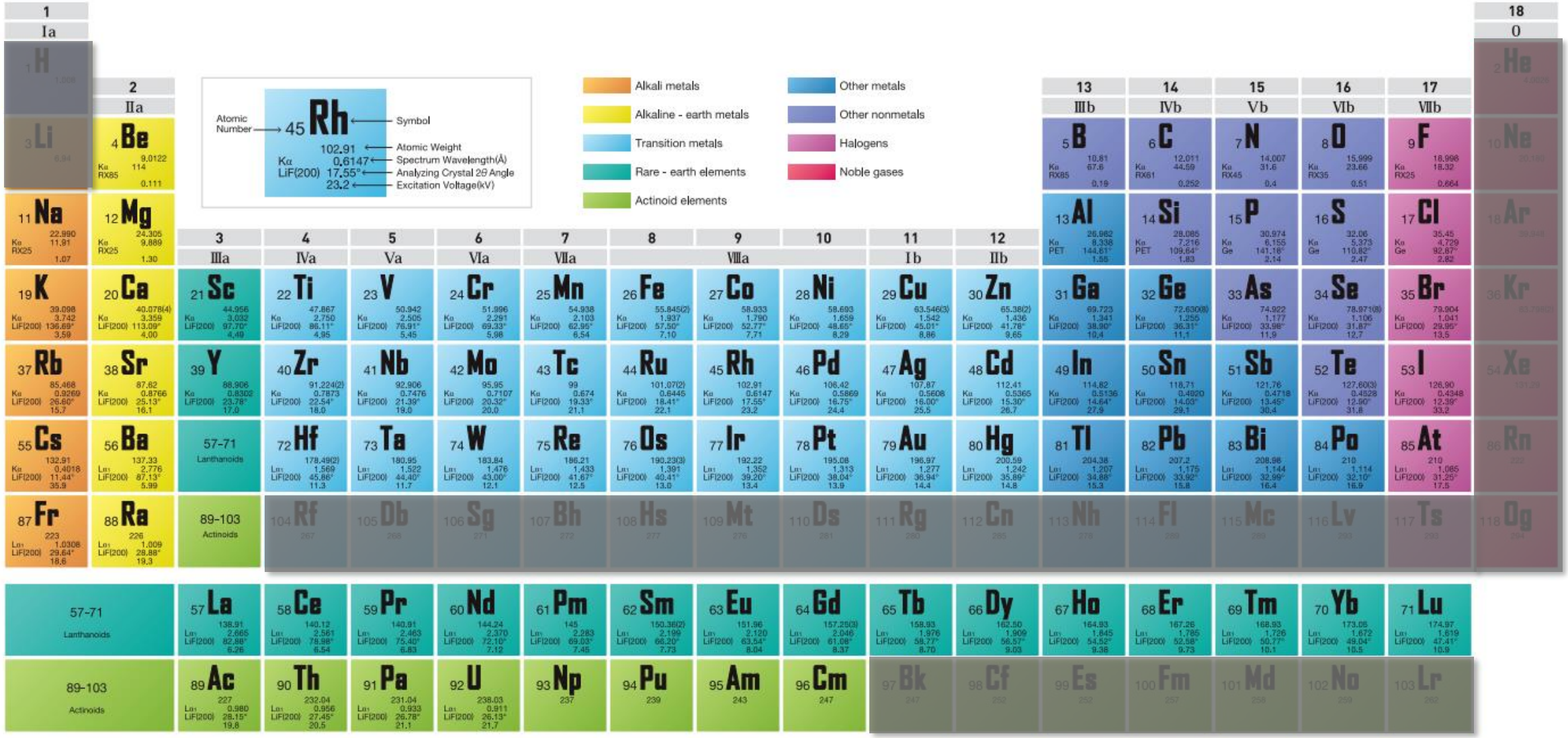
Sample Preparation: Overview



Sample Preparation: Pressed Pellet



Wide Range of Analytes



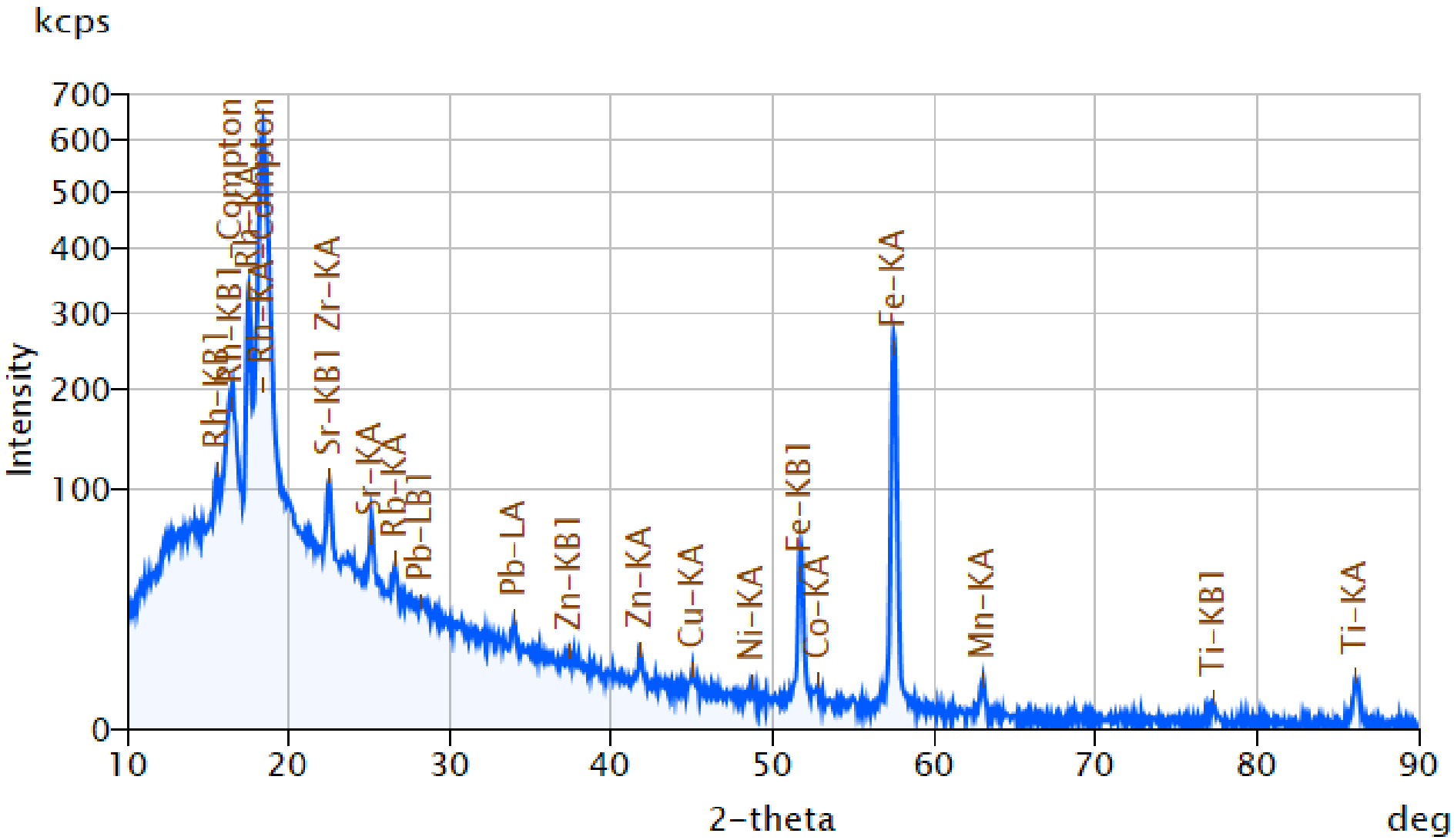
Ease of Use

Technique	ICP-MS	XRF
Prep time	Hours +	5-15 minutes
Prep materials needed	**Acids**, glassware, centrifuge, digesting block, hot plate, etc.	Press, die assembly
Analysis time	Minutes	<1-30 minutes
Sample recovery	Typically no	Yes
Calibration setup	Individual element	All elements
Calibration time	Hours +	Hour
Calibration upkeep	Daily +	Every 6 months
Operator	**Chemist**	**Anyone**

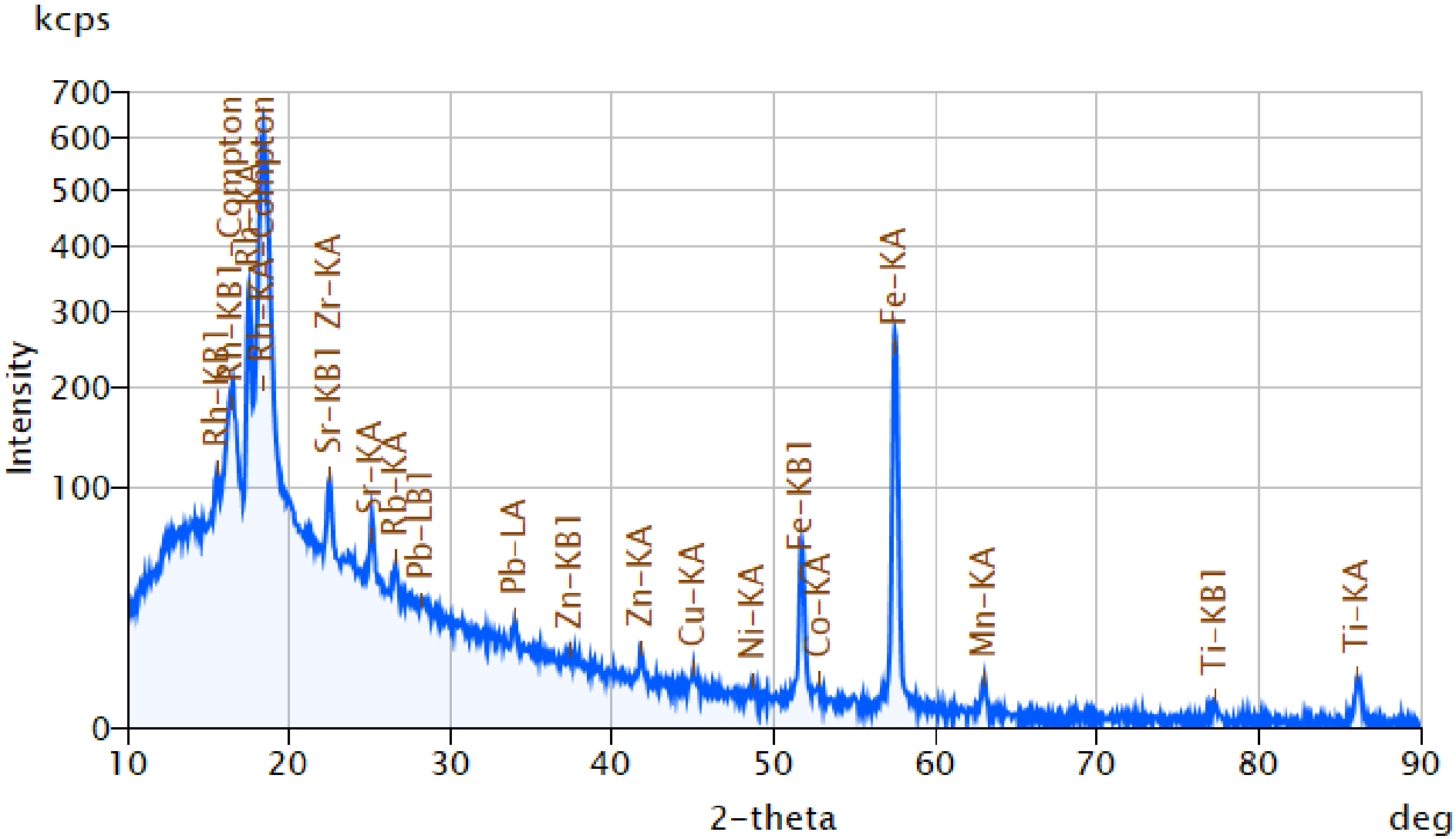
Types of Analyses

1. Qualitative (Scanning for all analytes!)
2. Semi-quantitative
3. Quantitative

Types of Analyses: Qualitative



Types of Analyses: Semi-quantitative



Comp.	Result (mass%)
Na2O	0.66
MgO	1.33
Al2O3	19.93
SiO2	42.09
P2O5	0.83
SO3	2.01
Cl	0.17
K2O	4.18
CaO	8.58
TiO2	2.91
MnO	0.25
Fe2O3	16.24
NiO	0.05
CuO	0.05
ZnO	0.09
Rb2O	0.07
SrO	0.18
ZrO2	0.13
PbO	0.20
SnO2	0.07

Types of Analyses: Quantitative

Natural Resources Canada | Ressources naturelles Canada

CCRMP
Canadian Certified Reference Materials Project

PCMRC
Projet canadien de matériaux de référence certifiés

Certificate of Analysis

First issued: April 2015 Version: April 2015

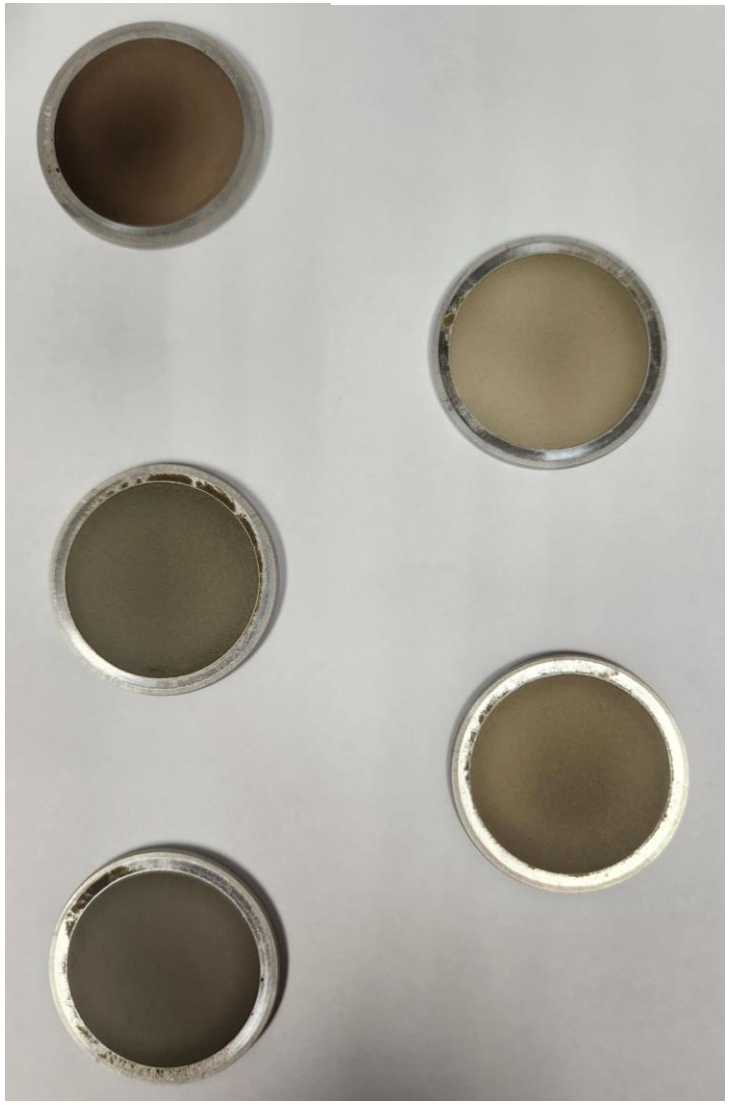
RTS-5

Certified Reference Material for a Nickel-Copper-Gold Tailings

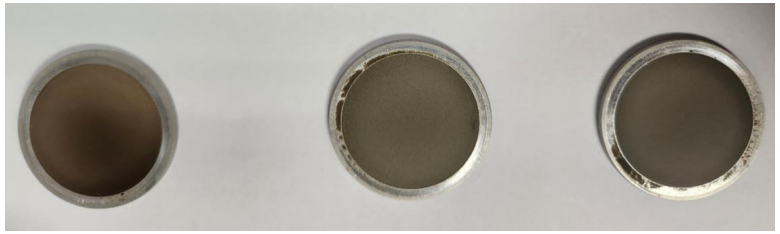
Table 1 – RTS-5 Certified Values

note: For some elements, some methods do not produce a complete recovery. Where possible, certified, provisional or informational values are provided for those cases.

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	1.50	0.07	0.10	0.04
Al (FUS) [a]	%	6.25	0.04	0.13	0.08
As	µg/g	1286	27	84	28
Au (no AD2) [b]	µg/g	0.408	0.017	0.017	0.007



Types of Analyses: Quantitative



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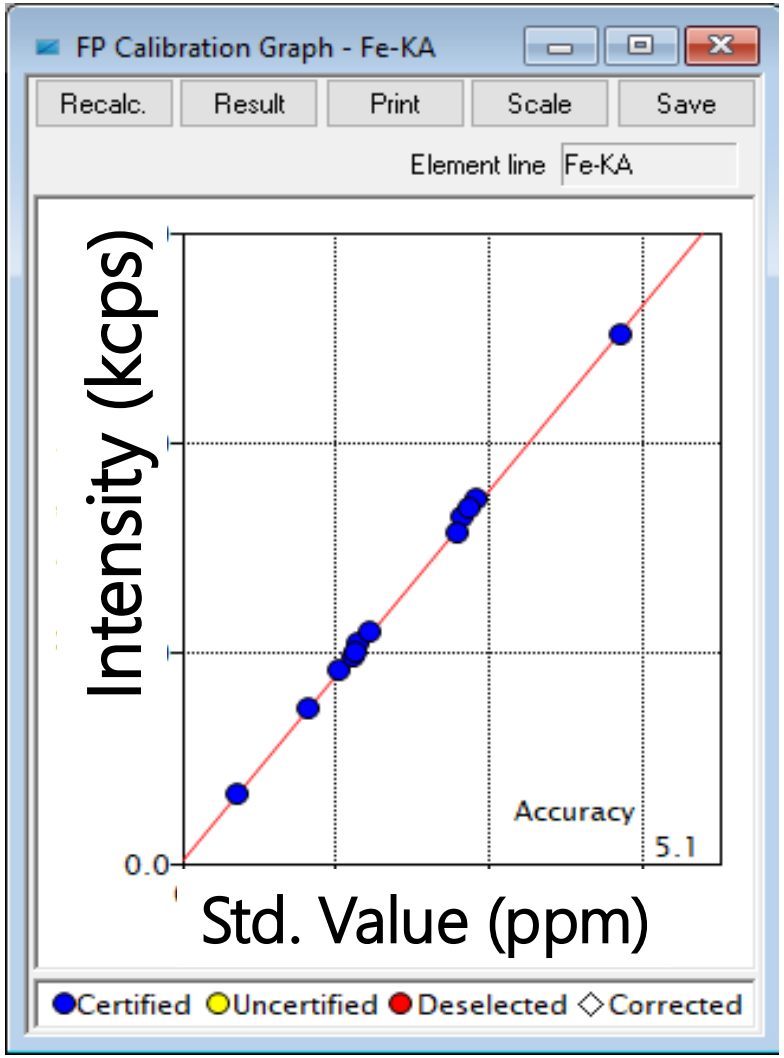
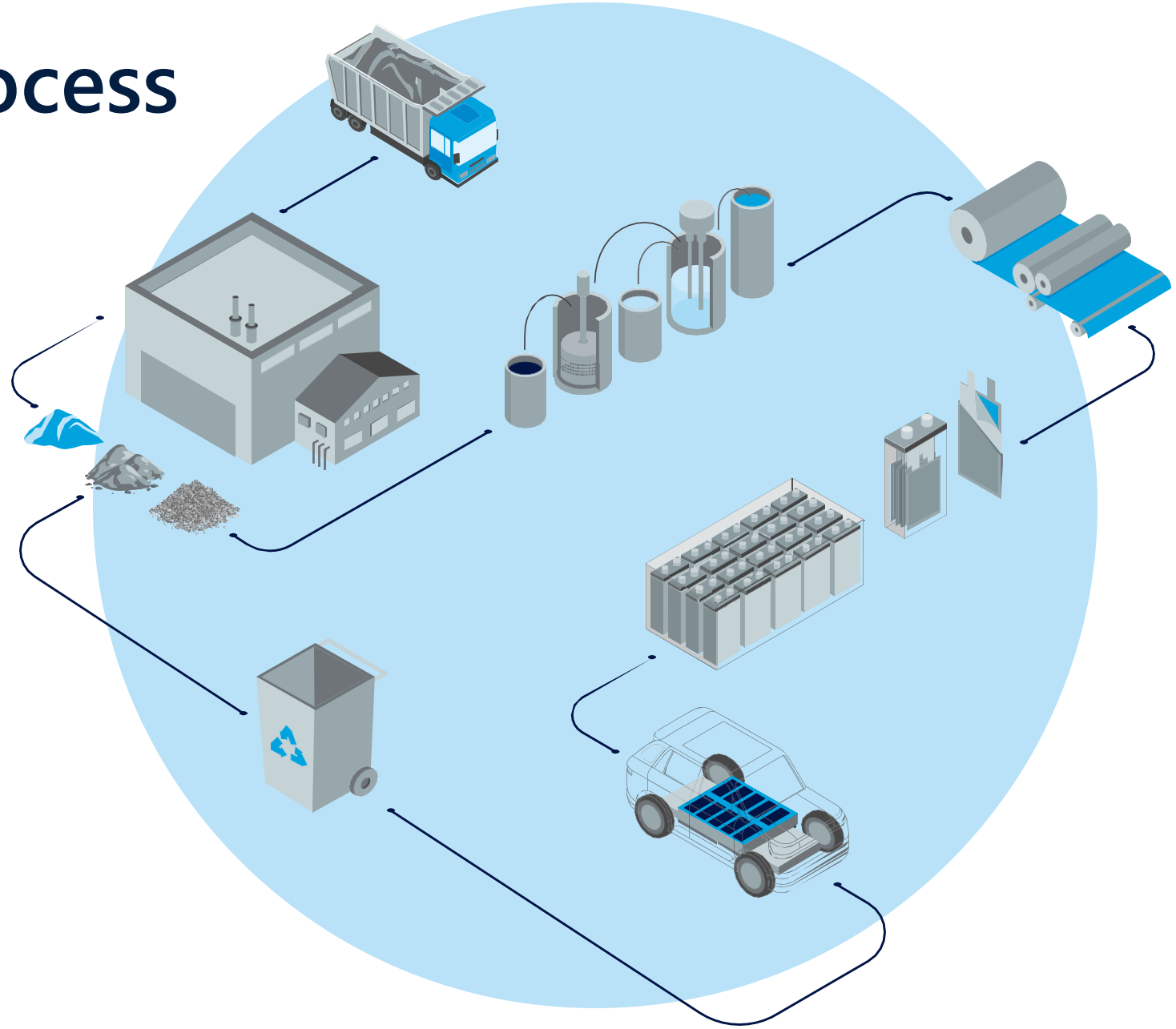


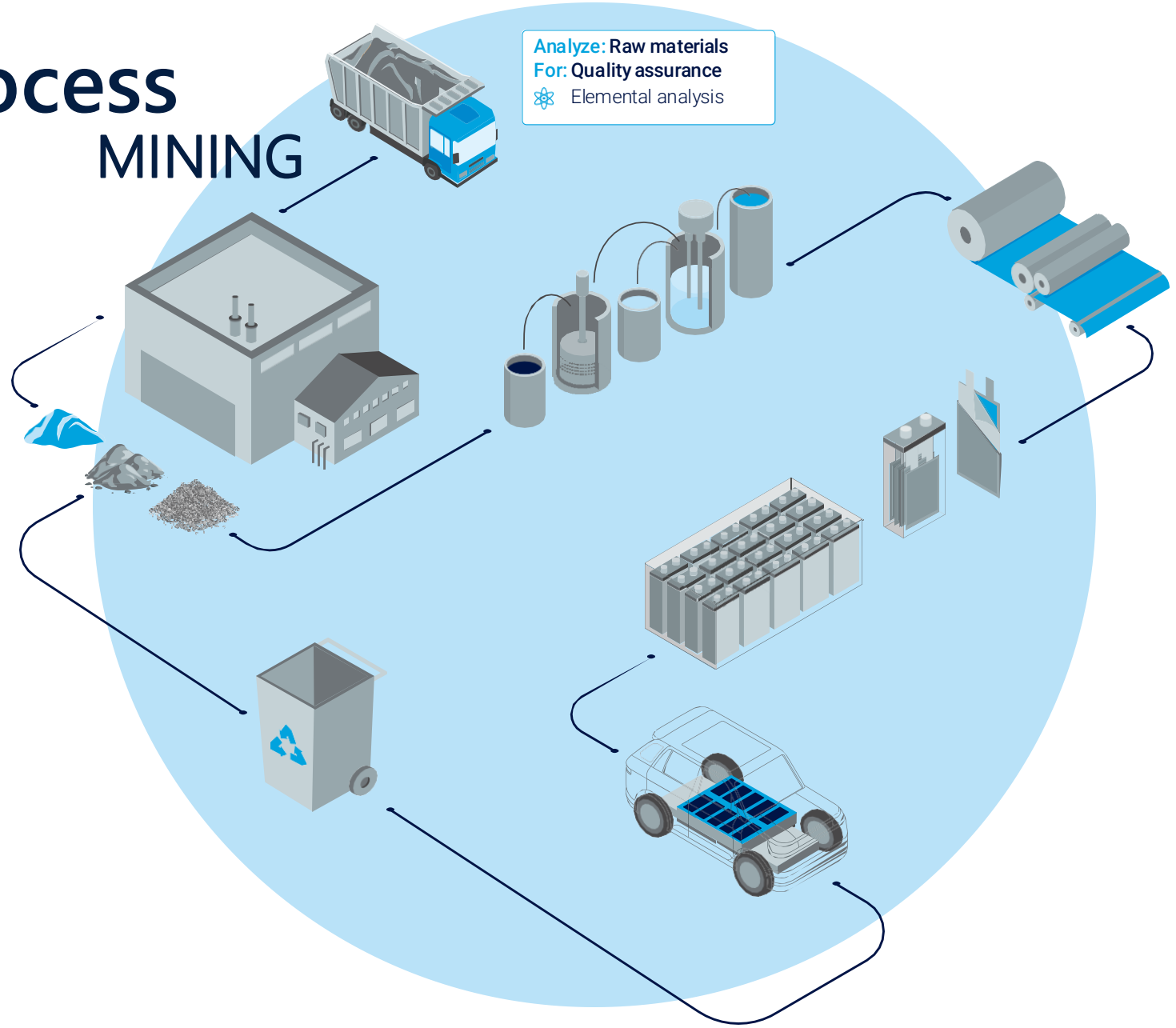
Where is XRF used?

The Process

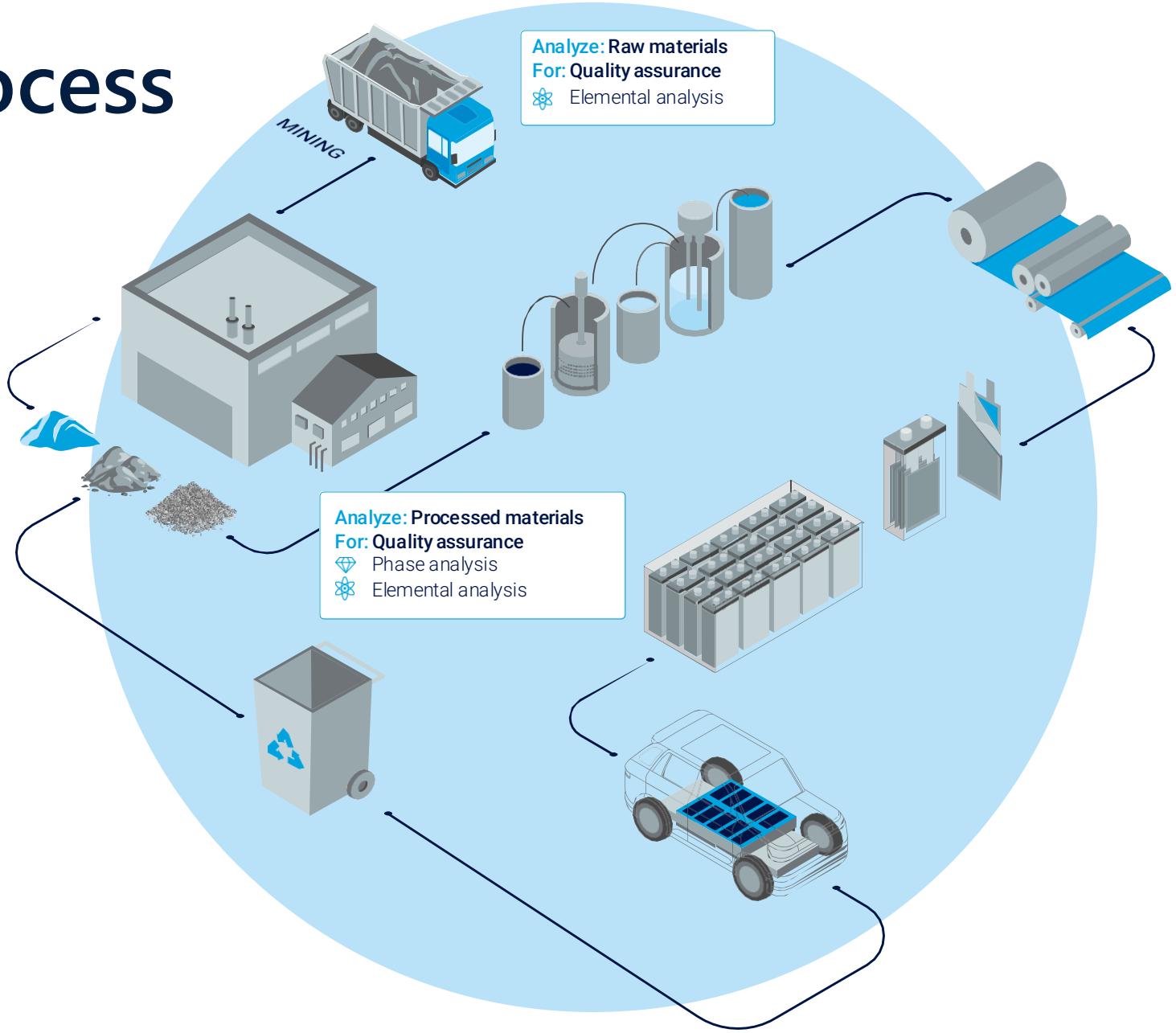


The Process

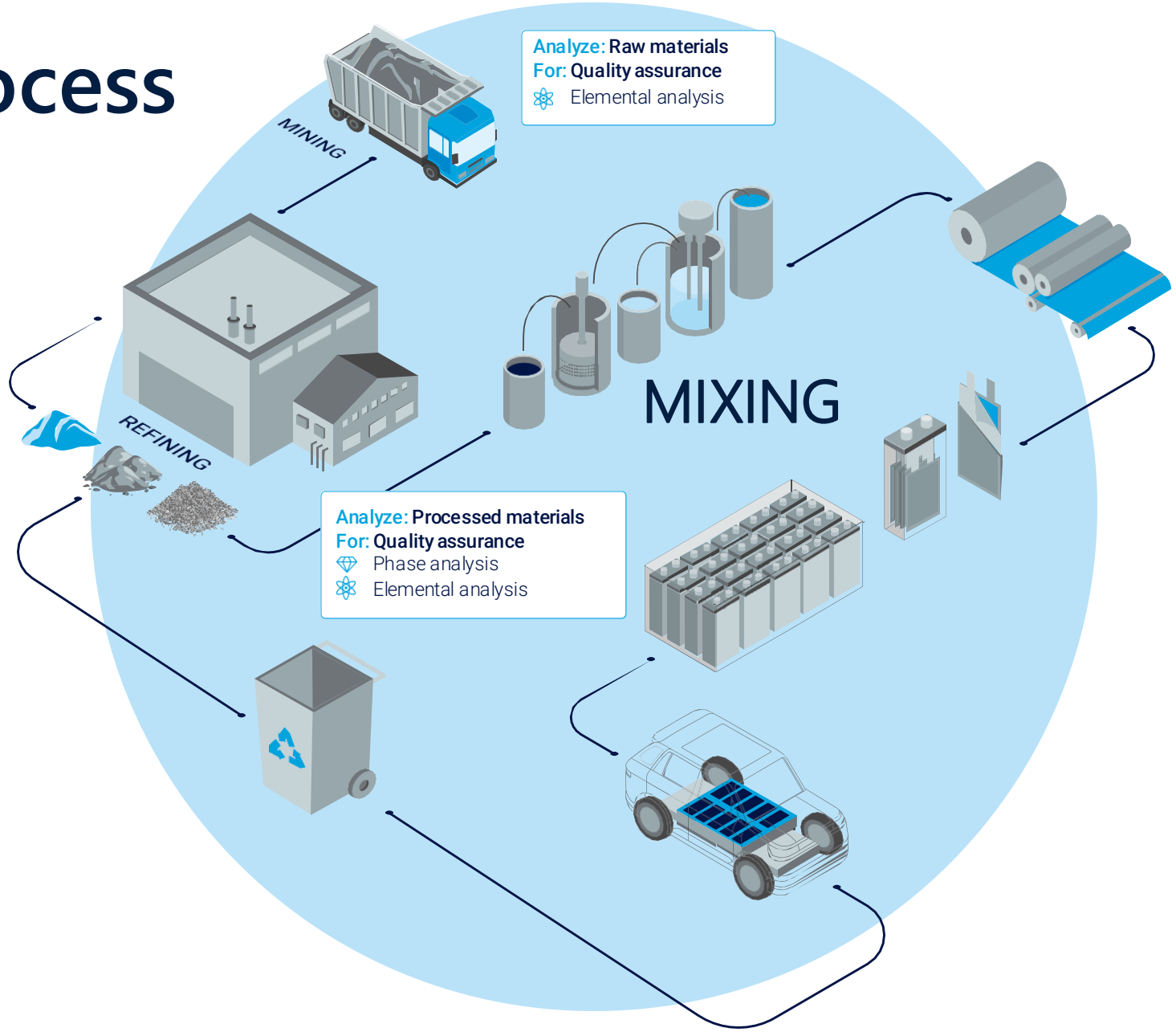
MINING



The Process



The Process



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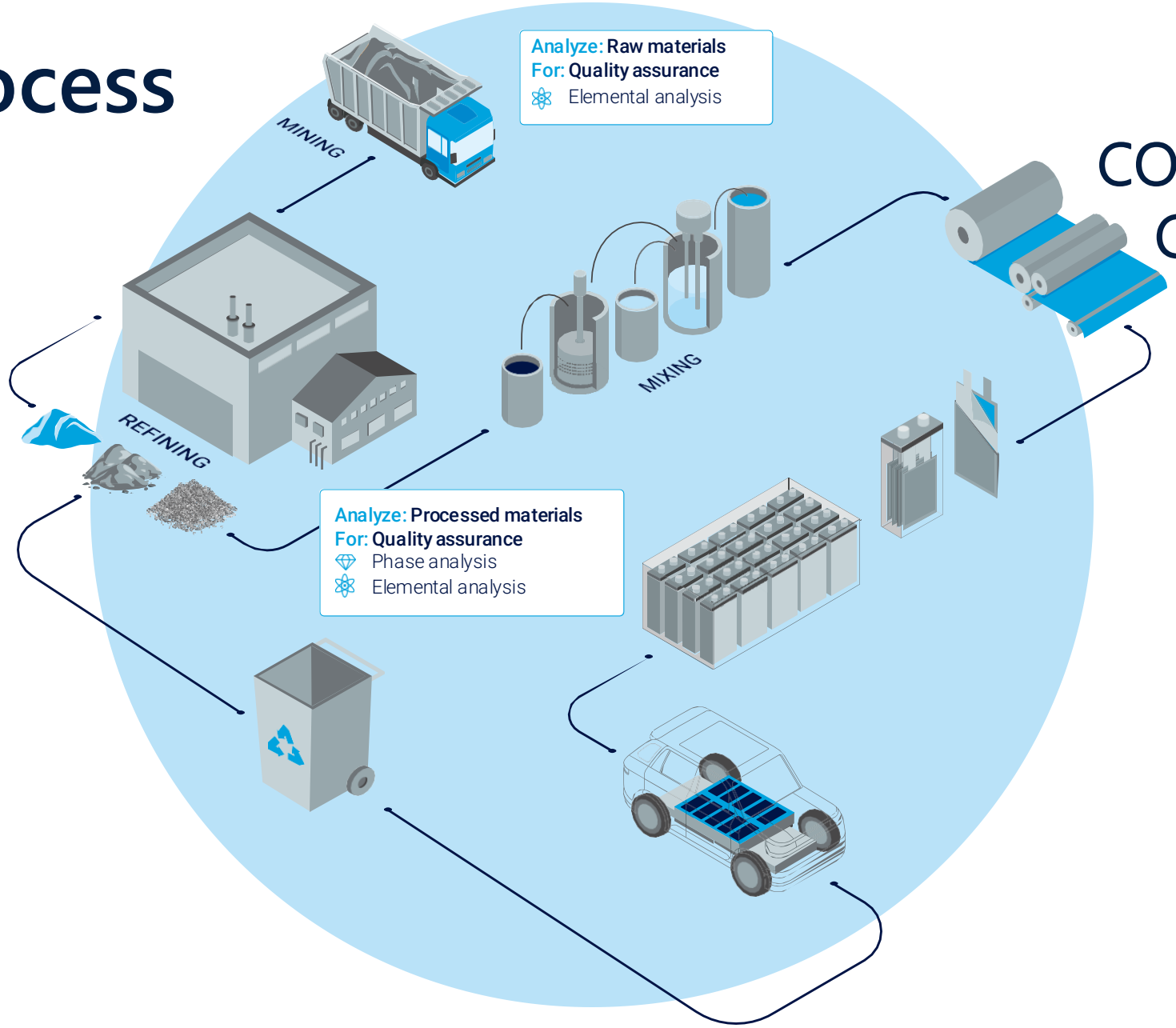
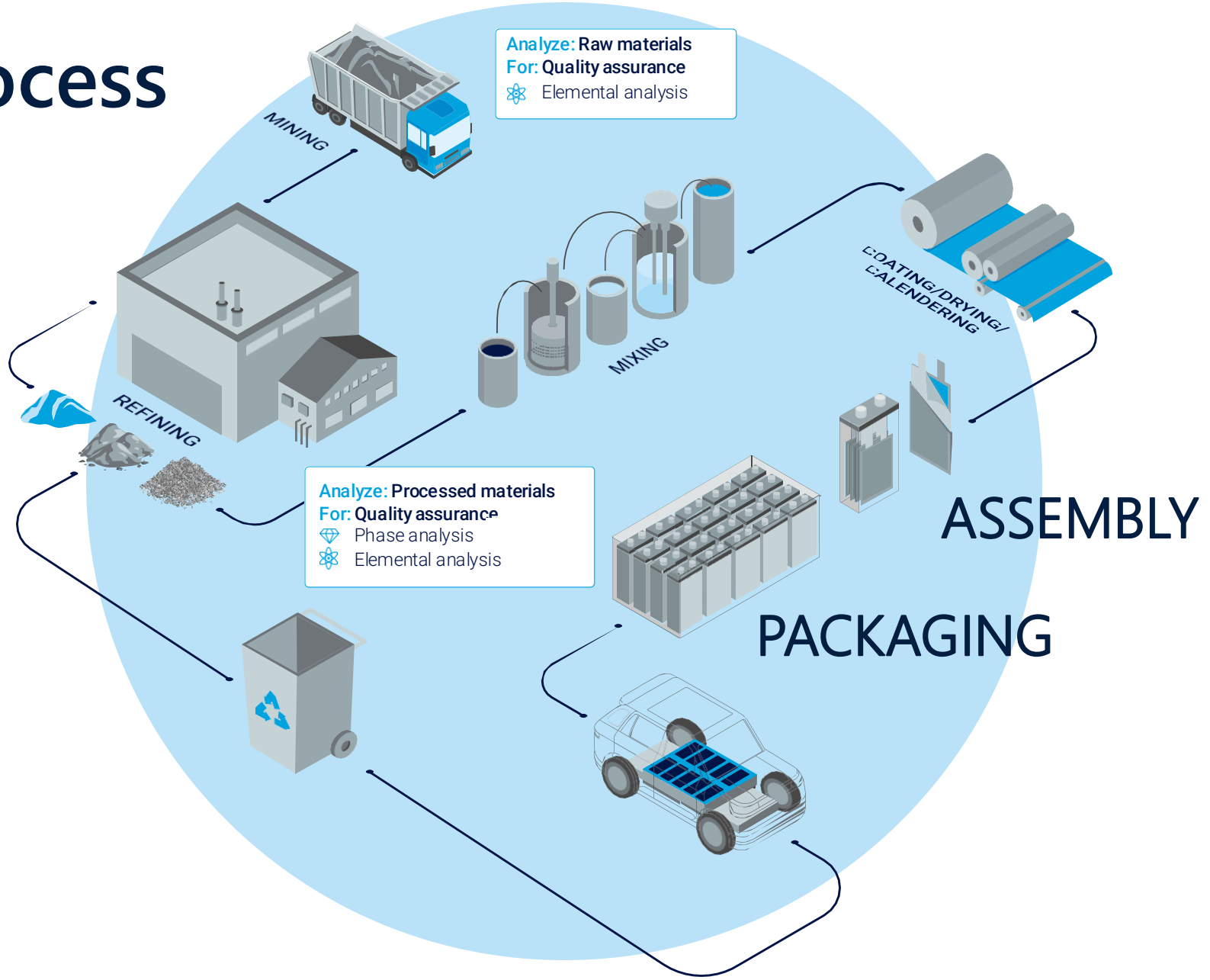


The Process



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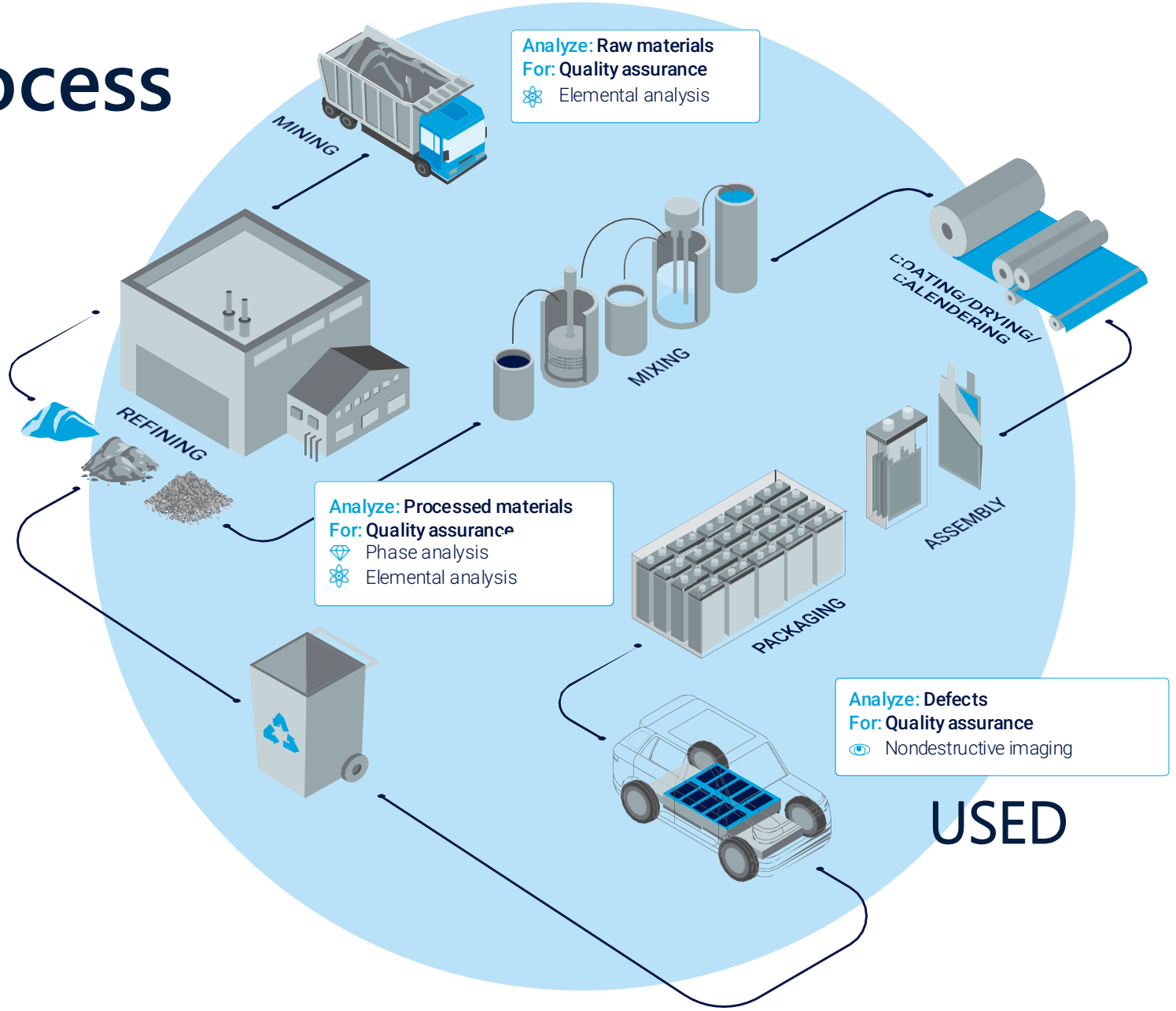
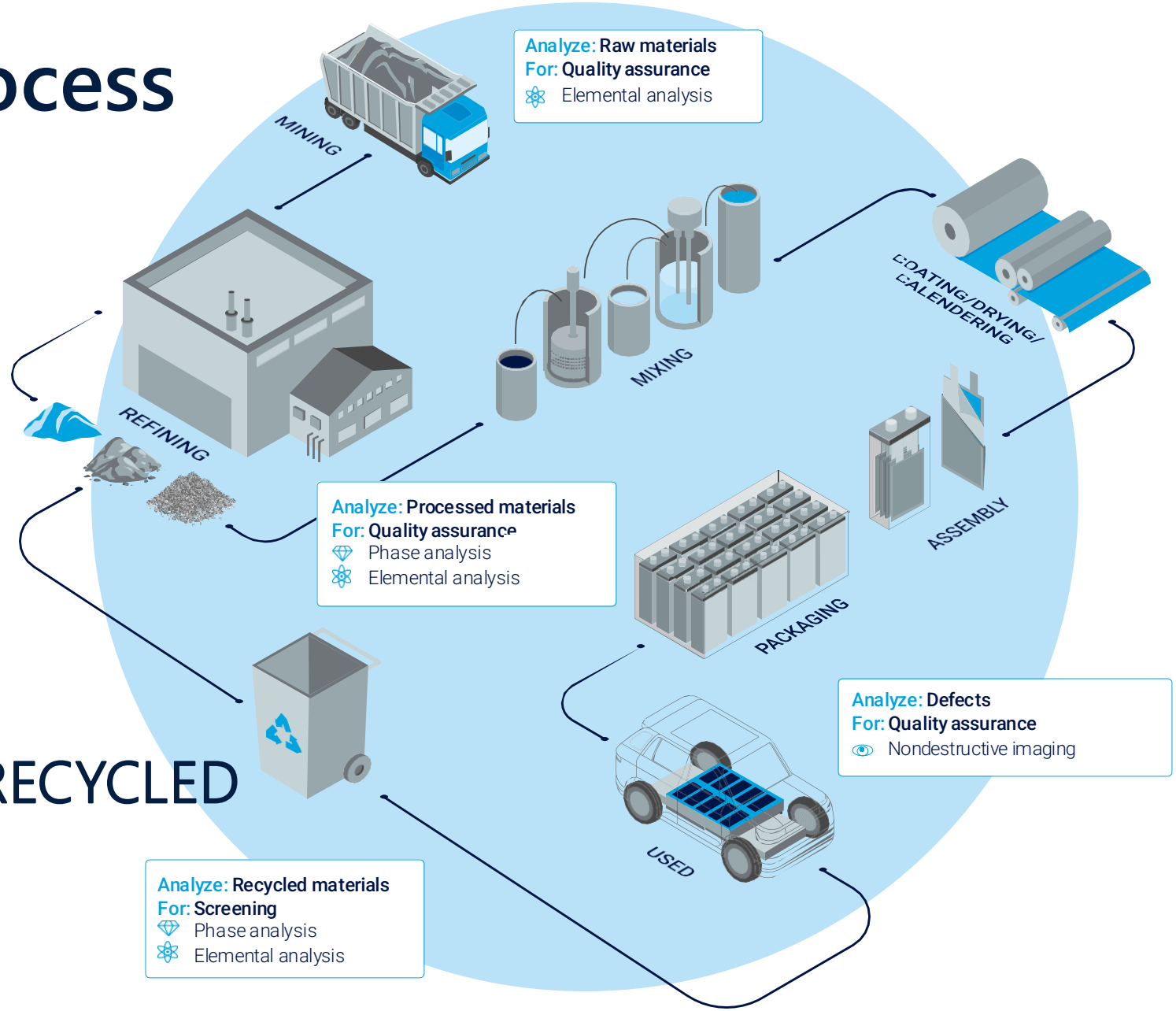


The Process



How is XRF used?

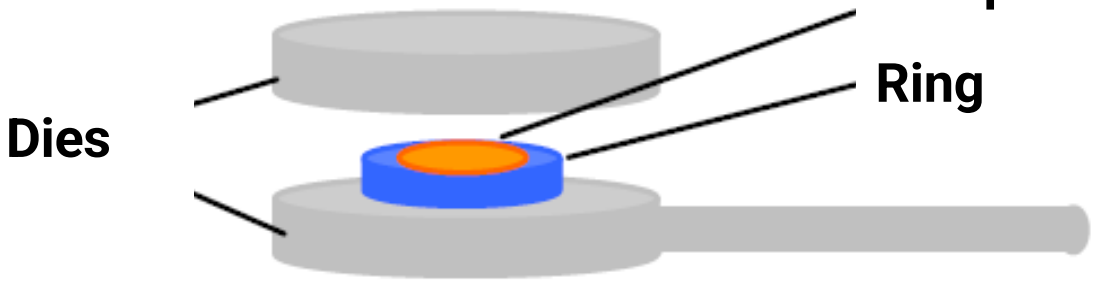
NCM Cathode Example

NCM Cathode Example: Prep

Sample → **Mix binder**

10-20 mass%

NCM Cathode Example: Prep



Sample → **Mix binder** → **Press**

10-20 mass%

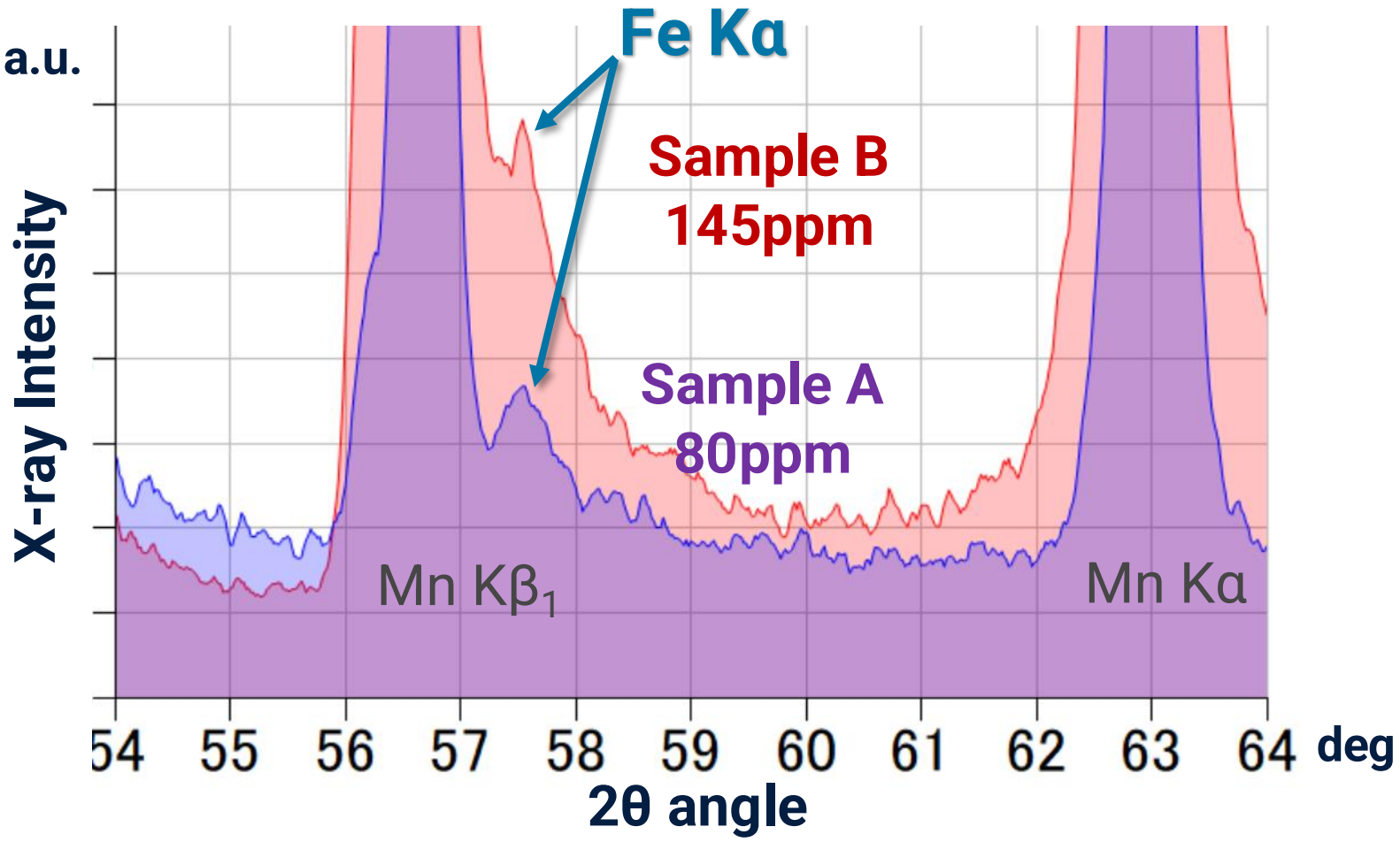
5-7g sample
32mm Al ring
10 Tons

NCM Cathode Example: Semi-Quantitative Results

		Ni	Co	Mn
Sample A	WDXRF	0.846	0.098	0.056
	ICP	0.85	0.10	0.05
Sample B	WDXRF	0.490	0.201	0.309
	ICP	0.50	0.20	0.30

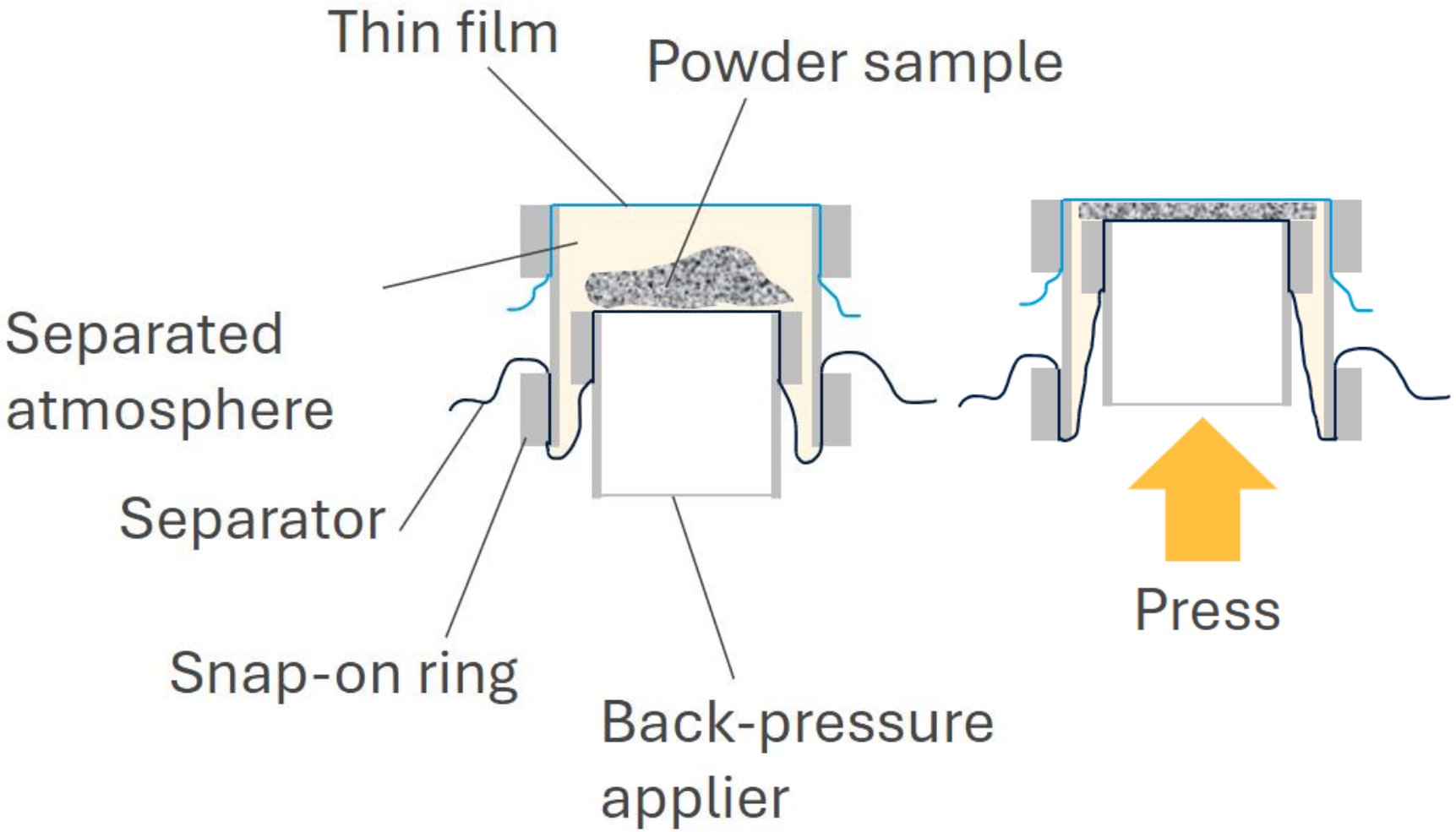
Unit : mol

NCM Cathode Example: Impurity Analysis

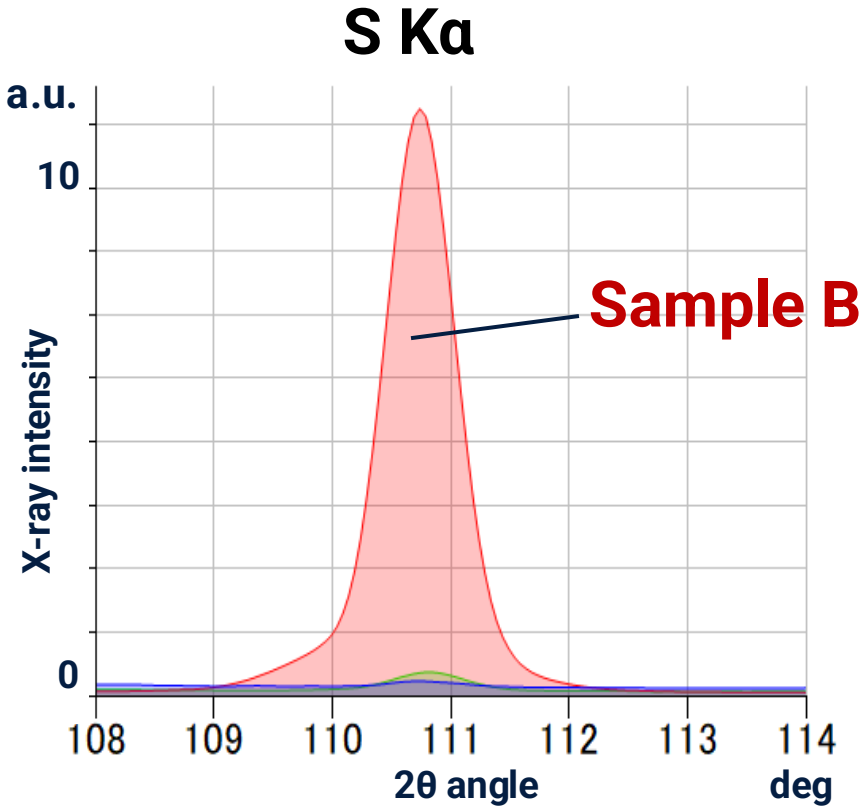


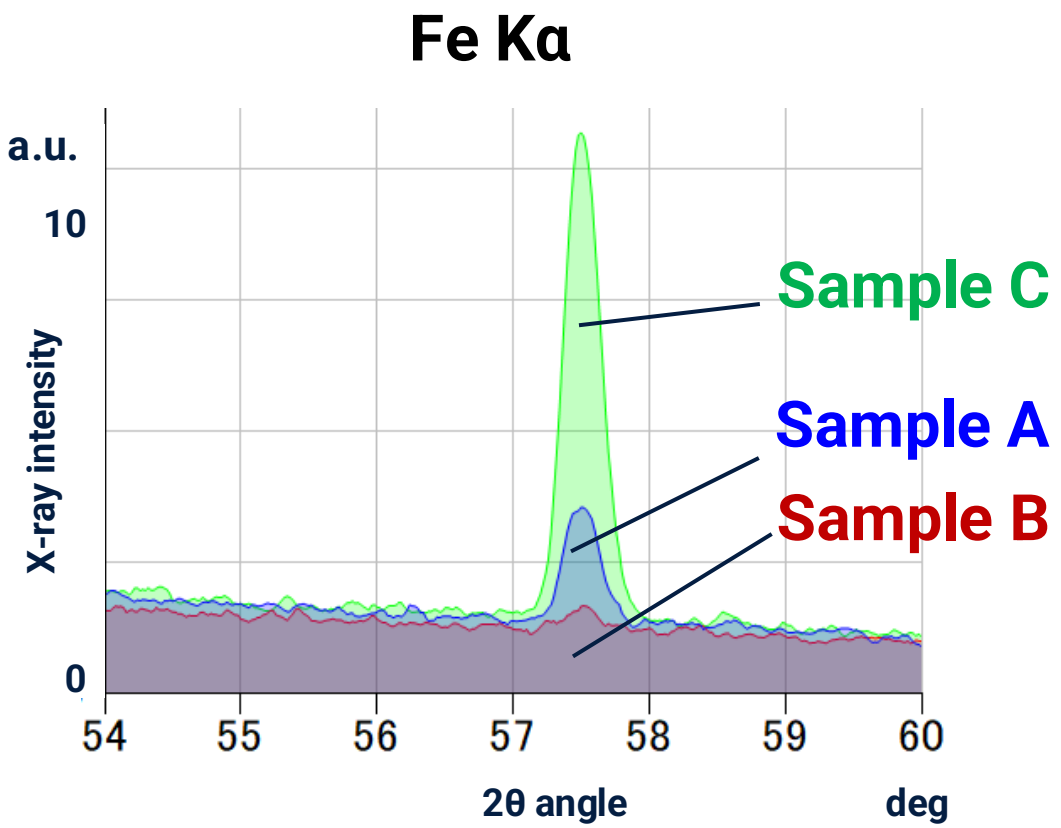
Graphite Anode Example

Graphite Anode Example: Prep



Graphite Anode Example: Semi-quantitative Results





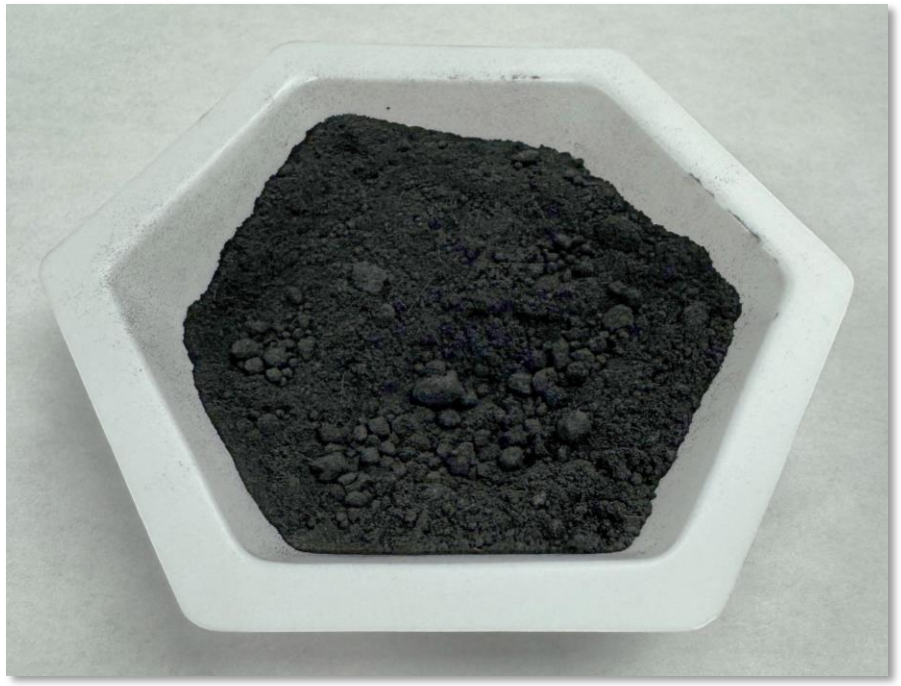
Graphite Anode Example: Semi-quantitative Results

	Na	Mg	Al	S	Ca	Fe	Zr
Sample A	83	249	28	38	73	39	13
Sample B	N.D.	975	24	2958	41	6	N.D.
Sample C	81	242	42	76	81	180	4

Unit : ppm

Black Mass Recycled Material Example

Black Mass Recycled Screening Example: Setup



NEX QC+
QuantEZ EDXRF

Black Mass Recycled Screening Example: Semi-quantitative Results

Element	Result (mass%)	Stat. error
Co	13.556	0.008
Mn	9.963	0.008
Ni	16.163	0.008
Al	6.156	0.009
Cu	6.605	0.009
Fe	2.104	0.003
Si	0.7577	0.0020
P	2.099	0.002
S	0.1275	0.0004
Cl	0.0317	0.0002
K	0.2074	0.0067
Ca	0.4066	0.0050
Ti	0.4774	0.0030
V	0.0527	0.0012
Cr	0.0382	0.0008
Zn	0.3678	0.0013
As	0.0028	0.0002
Zr	0.1358	0.0003
Ag	0.0026	0.0001
Cd	0.1214	0.0004
Sn	0.0766	0.0003
Sb	0.0256	0.0003
Ba	0.0248	0.0006
Pb	0.0528	0.0005

Polling Question #3

Microsoft Stock

Summary

1. What is XRF?
   - Elemental analysis covering a wide range
   - Variety of instruments
   - Variety of analysis “flavors”

Summary

2. Why use XRF?

- Non-destructive
- Ease of prep and use
- Variety of preparations to meet any needs
- Speed of analysis

Summary

3. Where is XRF used?

- Mining
- Refining
- Recycling

Summary

4. How is XRF used?

- Black mass recycling screening
- Graphite anode
- NCM cathode powder
- Many more!!!

Questions and Answers

We'll follow up with your questions.

Recording will be available tomorrow.

Check out our new Ebook!

Thank you!